Synthesis and Characterization of $Zn\text{-}Al_2O_3$ Composite

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
In
Metallurgical & Materials Engineering

By

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SAMBAL SHASHANK AMBU (109MM0101)

Department of Metallurgical & Materials Engineering
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CERTIFICATE

This is to certify that the thesis entitled, "**Synthesis and characterization of Zn-Al_2O_3 Composite**" submitted by **Sabarna Maity (109MM0117)** and **Sambal Shashank Ambu (109MM0101)**, in partial fulfillments for the requirements for the degree of **Bachelor of Technology** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ABSTRACT

In this thesis, comparative study of zinc and zinc/alumina composite is done. Fabrication of Zinc-Alumina metal matrix composite is done by in-situ reaction and direct blending of Zn with alumina was investigated. In in-situ process ZnO and Al were mixed and heated at 500^{o}C to produce two composites, Zn-48.45 vol. % Al_2O_3 and Zn-60 vol. % Al_2O_3. The same compositions were also developed by direct reaction. The composite developed was characterized using x-Ray diffraction, scanning electron microscope (SEM), hardness measurement using Vickers hardness tester, thermal property analysis and wear testing. Microstructural study of fractured samples were also done. The results show that reaction of ZnO and Al led to the displacement reaction to produce Al_2O_3 in small proportions. The reaction product microstructure also consist some nano-sized particles. Although the preparation of the composite by direct reaction does not show any sign of formation of nano-sized alumina. The wear properties and hardness value of composites are greatly superior than that of Zn metal. In fracture analysis of the composite it was observed that fracture surface is covered with alumina particles.

Contents

List of figures

Chapter 1

Introduction

1 Introduction

The composite materials are created by comprising two or more materials. The initial materials have mutually different properties and the newly formed compound gives entirely new set of properties.[1] The advantage of the composite material with respect to their substrates reflects in improvement of mechanical properties primarily in increase of specific elastic modulus, increased hardness, improved wear properties as well as resistance to corrosion.[2] The zinc based alloys have found considerably industrial use because of their excellent fluidity, castability and superior mechanical properties.[3] Despite the attractive room properties zinc alloys show unsatisfactory performance due to their low melting point

Reinforcing the stiffer, harder alumina matrix with ductile zinc metal/ zinc metal alloys provide a combination of properties of both metal and reinforcement.[4] In recent years many researchers have paid attention to new fabrication techniques such as in-situ and high energy ball milling (specially for nano-sized particles).[5]

In fact, Zn–Al_2O_3 composite can be prepared in two ways:

1. by direct addition of fine Al_2O_3 particles to Zn matrix; or
2. by a displacement reaction of Al with a ZnO according to the following reaction

$$3ZnO + 2A1 = Al_2O_3 + 3Zn \text{ [2]}$$

In first method Sintering of the direct blend of fine powders of Zn and is done. Sintering is a solid-state powder processing technique involving diffusion mechanism by heating below the melting temperature which results in uniform distribution of reinforcement in the matrix itself by the means of compaction. It is a simple and useful solid-state technique to synthesize both equilibrium and non-equilibrium material from initial powder [6, 7]. This technique can also be used for preparing homogenous composite particles with intimately dispersed uniform internal structure [8].

The second method of preparing the composite is the sintering of ZnO and Al. In this fabrication of Zinc Matrix Composite, reinforcing phase is formed in the matrix as a result of precipitation from the melt during its cooling and Solidification. There are many types of reinforcements that can be formed using in situ process. Here we have keen interest in synthesizing the dispersed phase in the form of particulates. In sinter-

ing synthesized particles smaller than those in materials with separate fabrication of dispersed phase (ex-situ MMCs). Even some times Nano-sized particles can also be fabricated. Fine particles provide better strengthening effect.

The thermal energy generated during sintering, has been found to facilitate chemical reactions leading to the metallothermic reduction and resulting in the formation of compounds.

When higher levels of Al_2O_3 are desired in the process, a larger amount of Zn is inevitably presented, thus it is conceivable that the deterioration of a material's properties cannot be avoided on account of the larger amount of Zn [9]. To optimize the properties, the existence of zinc must be taken into account, that is, besides designing the ceramic reinforcement, the matrix must also be designed.

Aluminum (Al) is a possible candidate to produce Zn- Metal Matrix Composite because the reaction between Al and ZnO has a high negative free energy change (ΔG) .

Alumina has a low cost which is why it is very economical to use alumina. Alumina can be found in a number of phases. Among all the phases α-Al_2O_3 is the only form of alumina which is stable. α-Al_2O_3 is chemically very stable and is highly unreactive. The stability of α-Al_2O_3 phase is also retained even at high strength at elevated temperatures. Among all the phases α-Al_2O_3 has the highest strength. Besides this alumina has very good high temperature properties, compressive strength and very good wear resistance properties. High coefficient of thermal expansion and low thermal conductivity are its major drawbacks. α-Al_2O_3 has a hexagonal structure. Alumina is a hard wear resistant material having good thermal conductivity and high strength and stiffness values.

Al_2O_3 (3.95gm/cc) is almost half as light as Zn (7.14 gm/cc). As a result during liquid phase sintering Al_2O_3 floats in Zn. Al_2O_3 is a ceramic. Zn has a low melting point (419.6^{o}C) and this is the major drawback of Zn as it cannot be used in high temperature applications.

Chapter 2

Literature Survey

2 Literature Survey

2.1 What is a composite?

Composite materials (shortened to **composites**) are materials made of two or more constituent materials which has mutually different physical, chemical or both properties, that when combined, produce a material with different characteristics from the parent components. The individual components remain separate and distinct within the finished product [10].

Composites are formed by the combination of two different materials usually separated by a distinct interface. The properties of composites are enhanced as compared to the properties of its individual components. The two phases that make up a composite are known as reinforcing phase and matrix phase. The reinforcing phase is inserted in the matrix which mainly enhances mechanical properties of the matrix. The reinforcing phases usually found in composites have different geometry such as particulates, fibers or sheets and the matrix materials can comprises of polymers, ceramics or metals .

Typical engineered composite materials comprise:

- Building materials such as concrete
- Reinforced plastics such as glass-reinforced plastic
- Metal Composites such as aluminium matrix composite
- Ceramic Composites [10].

2.2 Why to use composites?

The supreme benefit of composite materials is strength and stiffness pooled with weightlessness. By selecting a proper blend of reinforcement and matrix material, manufacturers can produce properties that precisely fit the requirements for a particular structure for a specific purpose [11].

Lightweight

Composites are extremely lightweight, especially when compared to materials like metal. Often a composite structure will weigh 1/4 that of a steel structure with the same strength.

High Strength

Composite materials are tremendously strong, especially per unit of weight. For example the high tenacity structural fibers used in composites such as aramid and S-Glass, are of very high strength.

Corrosion and Chemical Resistance

Composites are exceedingly resistant to chemicals and will have very negligible chance of getting corroded. This is why the marine industry was one of the first to adopt the use of composites such as fibre glass..

Elastic

Fiber reinforced composites have outstanding elastic properties. On bending metal, it will yield or dent. However, when composites are bent, they have the tendency to naturally snap back into place. This feature is ideal for springs, and is why composites are used in car leaf springs.

Non-Conductive

Certain composites for those which are made with fiberglass, are non-conductive in nature. This is important because often a structure is needed that is strong, yet will not conduct electricity.

The right composites also stand up well to heat and corrosion. This makes them ideal for use in products that are exposed to extreme environments such as boats, chemical-handling equipment and spacecraft. In general, composite materials are very durable [12].

Another advantage of composite materials is that they can be moulded into complex shapes. Composites thus provide design flexibility.

The shortcoming of composites is usually the cost. Although manufacturing processes of composites are often more efficient, the raw materials are expensive. Composites will never totally replace traditional materials like steel, but in many cases they are just what we need because of their needed properties.

Its properties can be jotted down as follows:

- High specific stiffness and strength
- Low weight
- Fibre orientation with the direction of principle stresses
- Excellent environmental and corrosion resistance
- Very low coefficient of thermal expansion
- Improved vibration damping properties
- Good fatigue resistance
- Relatively simple repair of damaged structures
- Ease of manufacture of complex shapes
- Low cost tooling options for one-offs, etc.[13]

2.3 Classification of composites:

Composites can broadly be classified in two ways [10] ;

1. On the basis of matrix
2. On the basis of reinforcement or the dispersed phase

2.3.1 On the basis of Matrix:

The matrix phase, having a continuous character. Matrix is usually more ductile and less hard phase. It holds the reinforced phase and shares a load with it.[10]. On the basis of matrix composites can be categorized mainly in four ways:

1. Ceramic matrix composite
2. Polymer matrix composite
3. Metal matrix composite

2.3.1.1 Ceramic matrix composite:

Ceramic Matrix Composite (CMC) is a material comprising of a ceramic matrix pooled with a ceramic (oxides, carbides) dispersed phase[10]. By combining different ceramic matrix materials with special suitable fibers, new properties can be created and tailored for interesting technical fields. In CMCs, only fiber components are used that withstand the relatively high temperatures required for the production of ceramics, without significant damage [14]. Ceramic Matrix Composites are reinforced by either continuous (long) fibers or discontinuous (short) fibers. Monofilament fibers produce stronger interfacial bonding with the matrix material improving its toughness [10]. Other requirements to be met are long term high temperature stability, creep resistance, and oxidation stability.

2.3.1.2 Polymer matrix composite:

Polymer Matrix Composite (PMC) is a material comprising of a polymer matrix pooled with a fibrous reinforcing dispersed phase [10]. Polymer matrix composites (PMCs) are comprised of a variety of short or continuous fibers bound together by an organic polymer matrix Most commercially produced composites use a polymer matrix material. PMCs are very popular due to their low cost and simple fabrication methods [15].

The reinforcement in a PMC provides high strength and stiffness. The PMC is designed so that the mechanical loads to which the structure is subjected in service are supported by the reinforcement. The function of the matrix is to bond the fibers together and to transfer loads between them. Chief among the advantages of PMCs is their light weight coupled with high stiffness and strength along the direction of the reinforcement [16].

2.3.1.3 Metal Matrix Composite:

Metal Matrix Composite (MMC) is a material consisting of a metallic matrix combined with a ceramic or metallic dispersed phase. The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous [17]. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. In structural applications, the matrix is usually a lighter metal such as aluminum, magnesium, or titanium, and provides a compliant support for the reinforcement. In high temperature applica-

tions, cobalt and cobalt-nickel alloy matrices are common [18]. The reinforcement does not always serve a purely structural task (reinforcing the compound), but is also used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity. The reinforcement can be either continuous, or discontinuous [19].

2.3.2 On the basis of reinforcement or the dispersed phase:

The secondary phase is embedded in the matrix in a discontinuous form. This secondary phase is dispersed and usually stronger than the matrix and hence called the reinforcing phase [20].

They can be classified into three ways as:

1. Particulate Composites
2. Fibrous Composites
3. Laminate Composites

2.3.2.1 Particulate Composites:

A composite that consists of tiny particles of one material implanted in another material. The reinforcing component is embedded in a matrix to form the composite [21]. The particulates can be very small particles (< 0.25 microns), chopped fibers (such as glass), platelets, hollow spheres. In each case, the matrix acts as binding medium necessary for structural applications and the particulates provide desirable material properties [10]. They can be subdivided as follows:

1. Composites with random orientation of particles.
2. Composites with preferred orientation of particles. Dispersed phase of these materials consists of two-dimensional flat platelets (flakes), laid parallel to each other[11].

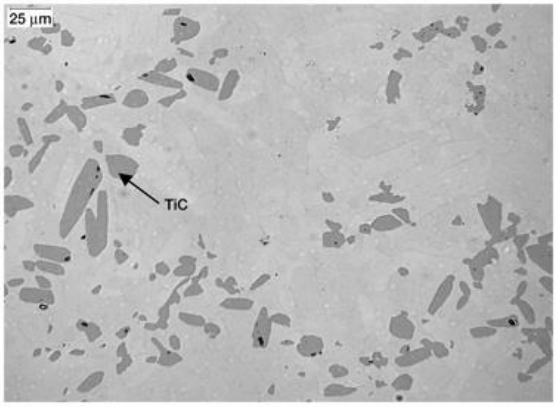


Figure 1: particles of TiC in Ti matrix [22]

2.3.2.2 Fibrous Composites:

Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers having a length < (100*diameter) and Long-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of continuous fibers. [10]. A fiber-reinforced composite (FRC) is a composite building material that consists of three components [23].

(i) the fibers as the discontinuous or dispersed phase,

(ii) the matrix as the continuous phase, and

(iii) the fine interphase region, also known as the interface.

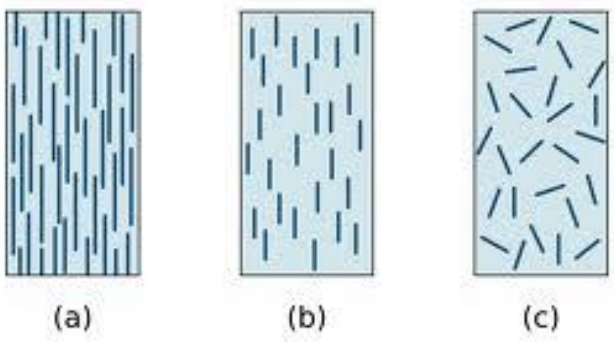


Figure 2 : (a) continuous aligned fibers (b) discontinuous aligned fibers (c) discontinuous fibers[23]

2.3.2.3 Laminate Composites:

When a fiber reinforced composite consists of several layers with different fiber orientations, it is called multilayer (angle-ply) composite[11]. Laminated Composites are made up of panels or sheets which are two dimensional in nature and the layers are arranged such that in each successive layer the orientation of the direction of high strength changes[13].

Figure 3: lamellar composite structure and fabrication[23]

2.4 Synthesis of Metal Matrix Composite:

There are many ways for the preparation of metal matrix composite

2.4.1 Solid state fabrication:

Solid state fabrication of Metal Matrix Composites is the process, in which Metal Matrix Composites are formed as a result of bonding matrix metal and dispersed phase due to mutual diffusion occurring between them in solid states at elevated temperature and under pressure.

Main two mechanisms working in solid state fabrication of MMC are diffusion bonding and sintering. Low temperature of solid state fabrication process depresses undesirable reactions on the boundary between the matrix and dispersed (reinforcing) phases [24].

There are mainly 3 ways for the fabrication of MMC by solid state route:

- Hot Pressing Fabrication of Metal Matrix Composites
- Hot Isostatic Pressing Fabrication of Metal Matrix Composites
- Hot Powder Extrusion Fabrication of Metal Matrix Composites[14]

2.4.2 Liquid state fabrication:

Liquid state fabrication of Metal Matrix Composites involves incorporation of dispersed phase into a molten matrix metal, followed by its Solidification. In order to provide high level of mechanical properties of the composite, good interfacial bonding (wetting) between the dispersed phase and the liquid matrix should be obtained. Wetting improvement may be achieved by coating the dispersed phase particles (fibers). Proper coating not only reduces interfacial energy, but also prevents chemical interaction between the dispersed phase and the matrix.[25]

There are many methods for the preparation of MMC by liquid fabrication route:

- Stir Casting
- Infiltration
- Gas Pressure Infiltration
- Squeeze Casting Infiltration
- Pressure Die Infiltration

2.4.2.1 Sintering:

Sintering fabrication of Metal Matrix Composites is a process, in which a powder of a matrix metal is mixed with a powder of dispersed phase in form of particles (generally powders) for successive condensing in solid state (sometimes with some presence of liquid) [26].

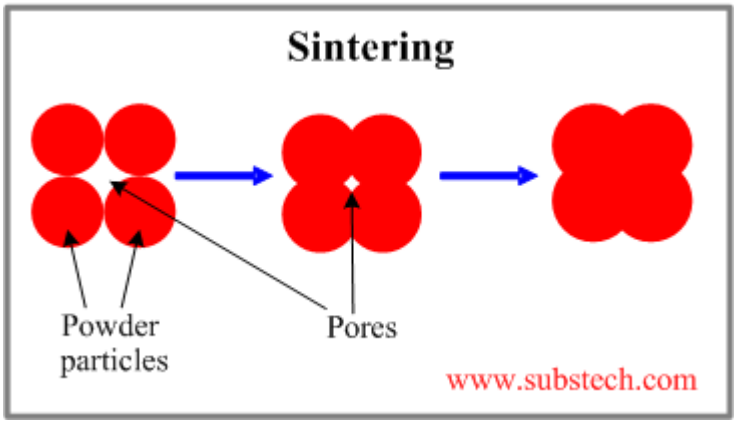


Figure 4: sintering mechanism [10]

Sintering is the method concerning merging of powderd materials by heating the "green" compact part to a high temperature below the melting point, where the material of the distinct particles diffuse to the adjacent powder particles. During the firing process, atomic diffusion drives powder surface exclusion in different stages, starting from the development of necks between powders to final eradication of small pores at the end of the process [27].

2.5 Wear Property:

Wear is the progressive loss of material from the operating surface of a solid occurring as a result of relative motion between two surfaces. Wear appears as if it occurs due to relative motion between two bodies which are solid. In most cases, wear is undesirable - it leads to increases in clearances between moving components, loss of precision, vibration and increased loading. Small losses of material due to wear can be enough to cause failure of large machines. However, in some cases high wear rates are desirable eg in grinding and polishing. The choice and surface treatment of materials in wear applications is very important . Even a surface that appears to be smooth, when magnified, can be seen to have many asperities (projections). Therefore when two surfaces meet, they contact only occasionally where the asperities happen to touch. The actual area of contact is therefore very small [28].

There are different forms of wear such as abrasion, adhesion (scuffing, wiping, galling), erosion and fatigue.

2.5.1 Types of wear:

1. Fretting wear
2. Corrosive wear
3. Adhesive wear
4. Fatigue wear
5. Abrasive wear

2.5.1.1 FRETTING WEAR :

Fretting wear is the repeated cyclical rubbing in between two surfaces, which is known as fretting, over a span of time which will eliminate material from one or both surfaces in interaction. An accompanying problem occurs when the small particles detached by wear are oxidised in air. The oxides are generally harder than the parent metal, so wear accelerates as the harder particles graze the metal surfaces further. In this process wear occurs by small amplitude vibrations at mechanical connections. Two fluctuating surfaces of metal when brought close to one another produce tiny metallic fragments that cause abrasion. There are different features that can affect fretting wear such as environmental circumstances, contact conditions and material properties. The different factors that can stimulate fretting are load frequency, number of cycles of fretting, humidity and temperature, etc.

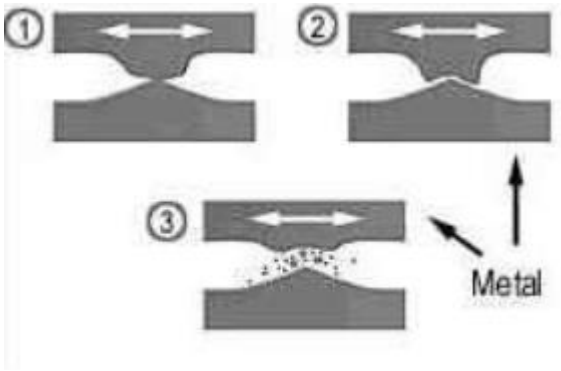


Figure 5: Schematic representation of fretting wear[23]

2.5.1.2 CORROSIVE WEAR :

Wear occurs in this method by the encouragement of corrosive agents . Here electrochemical exclusion of material occurs laterally with the removal of material through physical contact of the two surfaces.. These two phenomenons speed up the overall removal of material. The materials which generally resist the formation of oxides can be used in corrosive wear environments.

2.5.1.3 ADHESIVE WEAR**:**

Adhesive wear is the second most common form of wear in industry. It is defined as:

'The action of one material sliding over another with surface interaction and welding (adhesion) at localised contact areas' [29]

The name “adhesive” is given due to the development of a strong metallic bond between the asperities in the surface of the contacting materials [28]. Mild wear or oxidative wear occurs in the case where the thin oxide film on the surface inhibits the establishment of metallic bond between the asperities. Wear degrees are high for high load submission on the surface. This kind of wear is generally seen in sliding components in a valve, conveyor belts, fasteners, etc.

Severe wear occurs when the circumstances are such that the oxide is disordered at a greater rate than which it can reform, so that unsoiled metal is exposed below and massive adhesion occurs between the mating surfaces.

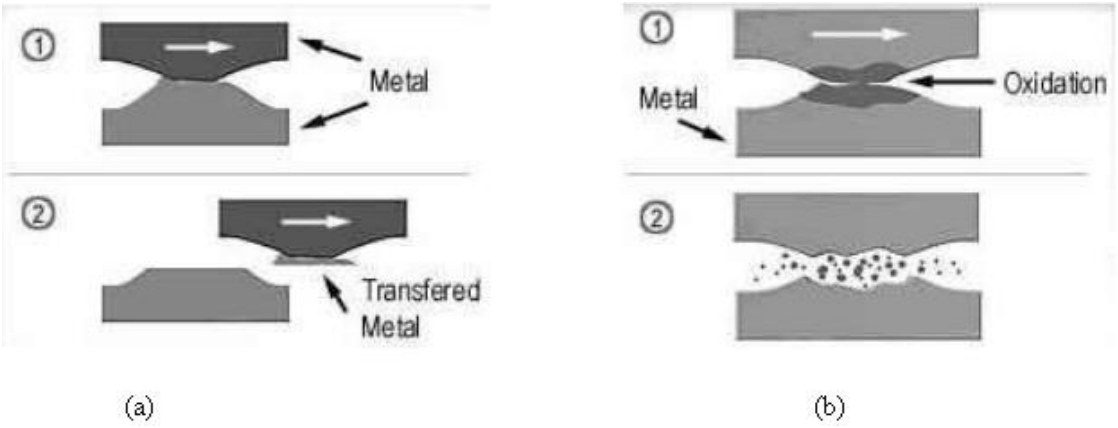


Figure 6: Schematic representation of (a) adhesive wear, (b) oxidative wear [23]

2.5.1.4 FATIGUE WEAR**:**

This type of wear can occur in surface of materials which are under cyclic stress. The extent of fatigue wear is determined by the factors like residual stress, surface appearance, microstructure and rigidity. Resistance to fatigue wear can be enhanced by surface treatments like carburizing, nitriding and shot peening [31].

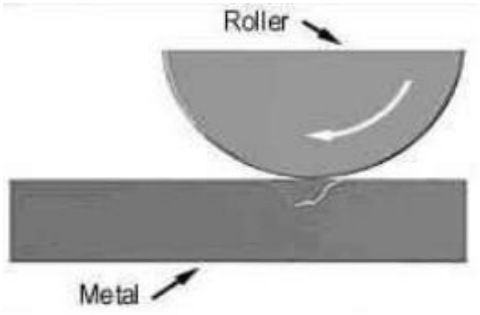


Figure 7: Schematic representation of contact fatigue [23]

2.5.1.5 ABRASIVE WEAR

In abrasive wear there is ploughing of localized surface contacts by a softer mated material [28]. If the particle is harder than the material then serious scratching or abrasion can occur. Abrasive Wear can be further subdivided into three types namely high stress, low stress and gouging. Aspects that can affect the resistance from abrasion are hardness, microstructure and for steel carbon content is also a factor [30].

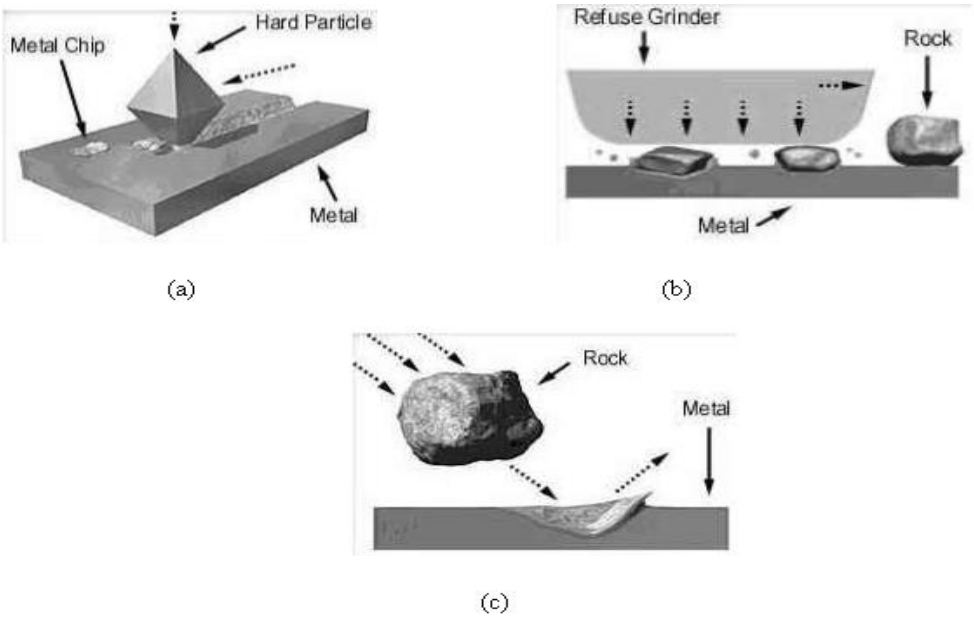


Figure 8: (a) Low stress abrasive wear, (b) high stress abrasive wear, (c) gouging [23]

2.6 Fracture Surface Analysis:

Fractography methods are routinely used to determine the cause of failure in engineering structures, especially in product failure and the practice of forensic engineering or failure analysis.[wiki] One of the aims of fractographic examination is to determine the cause of failure by studying the characteristics of a fracture surface In failure analysis, careful examination of the failed component, its design, its fabrication and its operating history can help to explain how and why a component failed. Failed parts are usually disposed without attempting to determine the causes of the failure. Because of these, failure rates and the costs associated with maintenance are high. The value of failure analysis lies in their ability to identify corrective actions by determining the causes of the failure. These corrective actions will minimize future failures of the same type. The failure analysis thus is a tool that can be used to reduce maintenance costs, increase system availability, and reduce the enormous potential losses involved in catastrophic failures. Failure analyses can also be used to identify the party at fault. Financial losses resulting from a failure can often be recovered when deficiencies in design, workmanship or materials can be identified [32].

Chapter 3

Experimental Procedure

3 Experimental Procedure

METHOD 1: BLENDING OF ELEMENTAL ZINC WITH ALUMINA

As received, powders of zinc and alumina were mixed to form two different mixtures having a total mass of 25g each such that the volume fractions of alumina in the mixtures were 48.45% and 60% respectively. Elemental zinc has been blended with alumina externally. This was done to achieve uniform distribution of the alumina particles throughout the zinc matrix.

METHOD 2: BLENDING OF ZINC OXIDE WITH ALUMINIUM

Powders of zinc oxide and aluminium were mixed to form two different mixtures having a total mass of 25g each such that the volume fractions of alumina in the mixtures were 48.45% and 60% respectively after the samples were heated.

These blended samples are then compacted using a die of diameter 12mm under a load of 300Mpa. Cold compaction machine was used to compact these powders.

Figure 9. Hydraulic Press Machine used for compaction

Now the compacted pellets of Zn and Alumina were taken and heated in a furnace in an inert atmosphere at 500^0C for two hours. In the same way pellets of ZnO and Al_2O_3 were heated at 800^0C for two hours. This was done to further improve the strength of the compacted powder samples. Tubular furnace was used for sintering of the pellets.

Figure 10: Tubular Furnace used for sintering

The heating rate used was 5^oC/min and the holding time for each sample was 1 hour after the heating period. The inert gas used for sintering was argon and its purity level was 99.999%. For comparisons along with these samples, pure zinc was green compacted and another same was sintered at 500^0C for 2 hours under same prevailing conditions.

After the sintering process, the samples were taken for XRD [33] analysis. The model of the XRD machine used was PW-3040. The scanning rate used was 3o per minute. The XRD data for all the samples was obtained in the form of graphs.

Figure 11. PW-3040 XRD instrument

Then the samples were taken for SEM [34] analysis. The instrument model used for the SEM analysis was **JEOL JSM-6480LV.**

Figure 12. JEOL JSM-6480LV scanning electron microscope

A potential of 15KV was used for the scanning electron microscope and the magnification used for the samples were varied. The size of the indication marker in the SEM graphs was 1 micron.

Then SEM data were taken and analysed.

Thermal Analysis [35] of Zinc-alumina composites prepared by the metallothermic reaction was done.

Then all the samples were taken for hardness measurement using Vickers hardness. The model of the apparatus used for discovery out the Vickers hardness was LECO-LV700. Hardness of all the samples were measured under a load of 5kg and noted down.

Figure 13. Vickers Hardness Testing machine

Wear testing of all the samples was carried out. Wear testing in terms of sliding wear resistance of all the samples was assessed using a ball on disc type wear testing apparatus The wear studies have been done at a load of 10N load for time of 10 min. at a rotational speed of 15 rpm and 4 mm track diameter. test. The results for the wear depth were obtained and the sliding distances were calculated by using the values of the wear depth and the process constraints. Graphs were plotted between the wear depth and sliding distance at a certain temperature. These graphs were analysed. The wear surfaces were once again taken for SEM analysis. The SEM images obtained for the wear surfaces of different samples were examined.

Figure 14. Wear testing machine

After the wear has been completed, the samples were splintered as to be compared under and then SEM was done to study and analysis the fracture surfaces.

Chapter 4

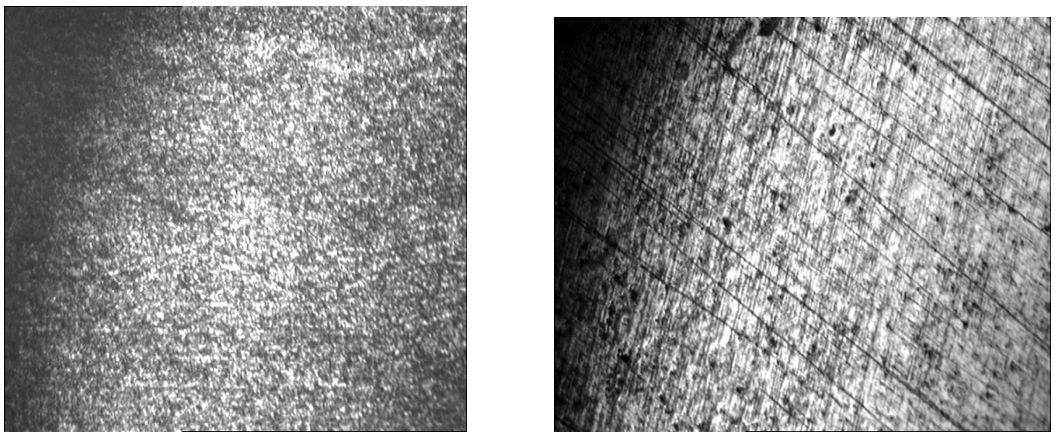
Results and Discussion

4 Result and Discussion:

4.1 Results of the sample prepared by direct blending of Zn and A2O3:

4.1.1 Optical Micrograph analysis:

4.1.1.1 green compact and sinters sample of elemental zinc:



a **b**

Figure 15 : Optical micrographs (a) green compact Zn (b) sintered Zn

4.1.1.2 Green compact and sinters sample of Zn-48.45 vol % Al_2O_3 :

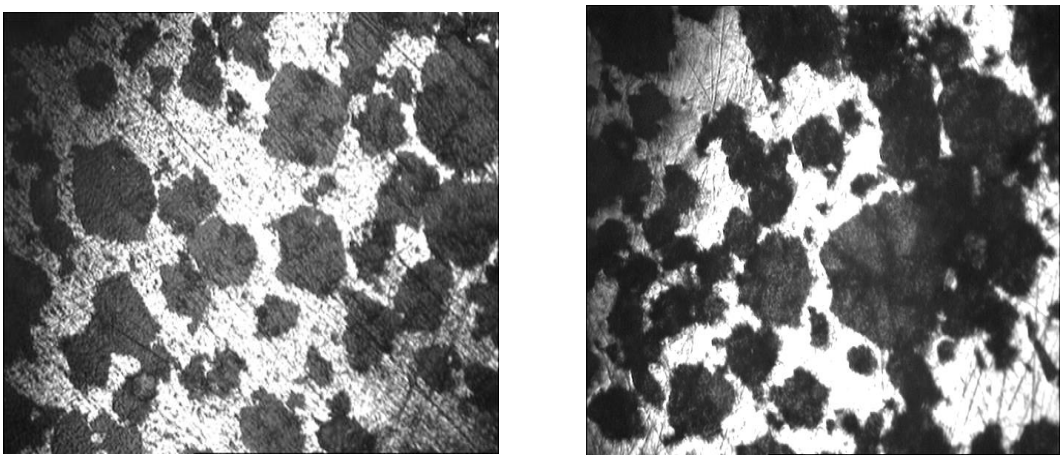


Figure 16: Optical micrographs (a) Zn-48.45 vol % Al_2O_3 *green compact 10x (b) sintered Zn-48.45 vol. %* Al_2O_3 *at 500oC for 2 h 10x.*

4.1.1.3 Green compact and sinters sample of Zn-60 vol % Al_2O_3:

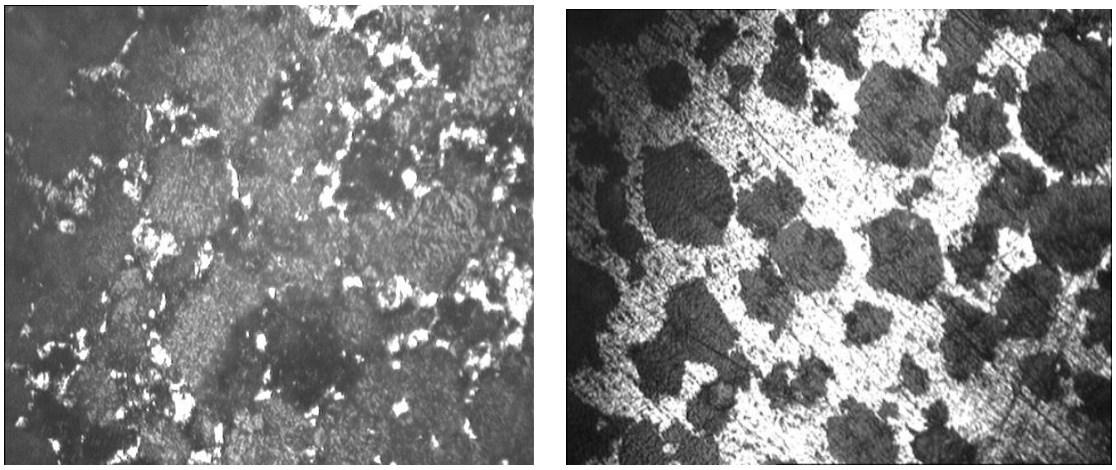


Figure 17: Optical micrographs (a) Zn-60vol% Al_2O_3 *green compact (b Zn-48.45 vol. %* Al_2O_3 *green compact at 10X*

The optical microstructure clearly shows that Al_2O_3 rich regions are surrounded by metallic Zn.

Al_2O_3 has a melting temperature of 2072^oC. At the sintering temperature of 500^oC molten Zn will be able to flow freely and surround the Al_2O_3 rich regions. At this temperature Al_2O_3 remains solid.

Zn has a density of 7.14 gm/cc and the density of Al_2O_3 is 3.95 gm/cc. As Zn has a higher density compared to Al_2O_3 it is possible that Al_2O_3 floats in molten Zn whereas Zn remains at the bottom. Due to the better mechanical properties of Al_2O_3 the properties like hardness and wear could be better at the surface of the compact compared to inside of the compact

This is why there is a lot of alumina on the top surface with the bottom layer containing more Zn. The top surface of the sample is expected to contain a large amount of Al_2O_3 as compared to the inner layer of the sample. Al_2O_3 is encapsulated by metallic Zn. The comparison of the sintered and unsintered sample suggests that the metal matrix consisting of Zn has become highly dense alumina rich regions have also become dense compared to the unsintered sample. There is some amount of Zn present in the Al_2O_3 rich regions.

4.1.2 SEM Images of Zn-60 vol % Alumina sintered at 500 C for 2 hrs:

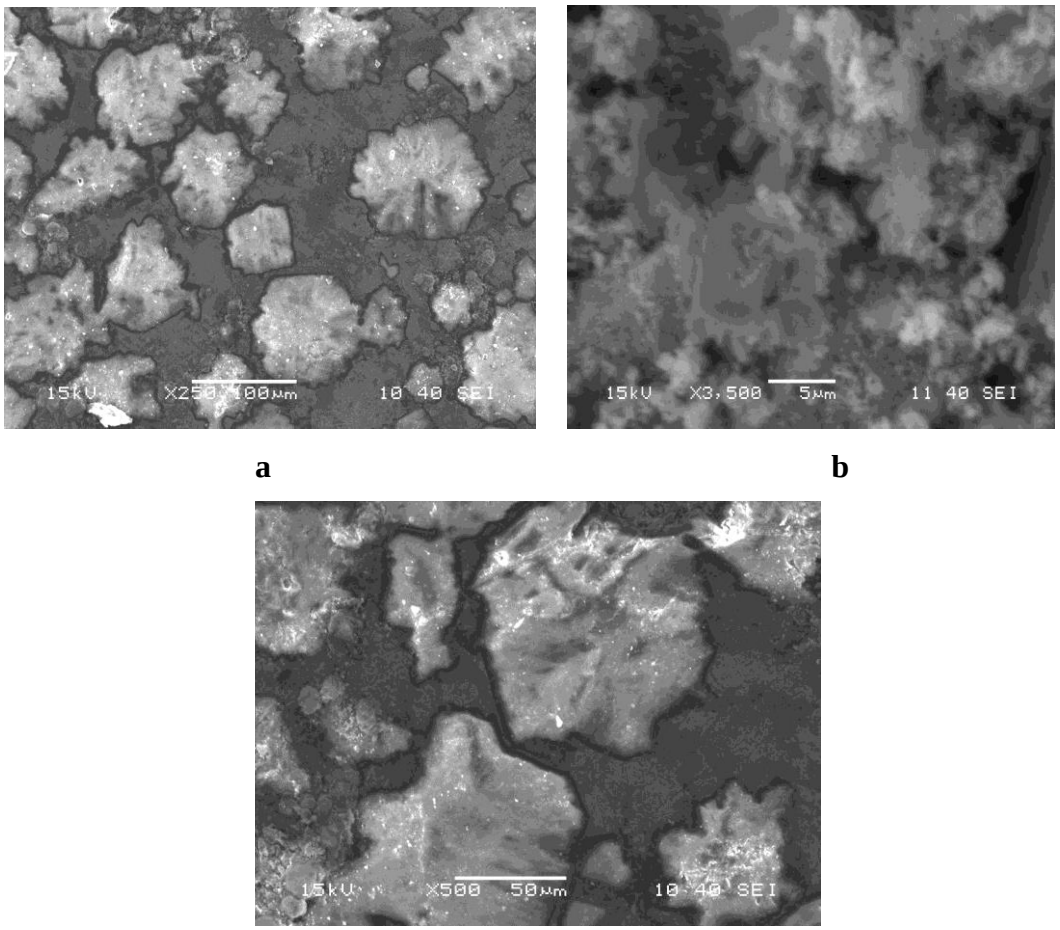


Figure 18: Zn-60 vol % Alumina sintered at 500 C for 2 hrs a) 250X b) 3500X c) 500X

The density of Zn is 7.14 gm/cc whereas the density of Al_2O_3 is 3.95 gm/cc.Alumina is much lighter than Zn. So in the molten Zn,Al_2O_3 will float. This is why there is a lot of white alumina on the top surface with the bottom layer containing more Zn. The top surface of the sample is expected to contain a large amount of Al_2O_3 as compared to the inner layer of the sample. Al_2O_3 is encapsulated by metallic Zn. The comparison of the sintered and unsintered sample suggests that the metal matrix consisting of Zn has become highly dense alumina rich regions have also become dense compared to the unsintered sample. There is some amount of Zn present in the Al_2O_3 rich regions.. In the higher resolution images, It can also be seen that formation of nano particle has not been taken place.

4.1.3 Hardness analysis:

Composite	Hardness (HV 5)
Zn (sintered at 500°C for 2h)	153.5
Zn-34.207 wt.% Al_2O_3 (sintered at 500°C for 2h) Zn- 48.45 vol. % Al_2O_3	302.75
Zn-60 vol.% Al_2O_3 (sintered at 500°C for 2h)	315

Table 1: hardness comparision of composite and elemental Zn

The hardness values indicate that the hardness of Zn-34.207 wt.% Al_2O_3 sample sintered at 500°C for 2 h is almost double the hardness of pure Zn samples sintered at the same temperature. But as we can see clearly that increment of alumina % to 60 volume % does not enhances the hardness much.

4.1.4 Wear Studies:

4.1.4.1 wear graphs:

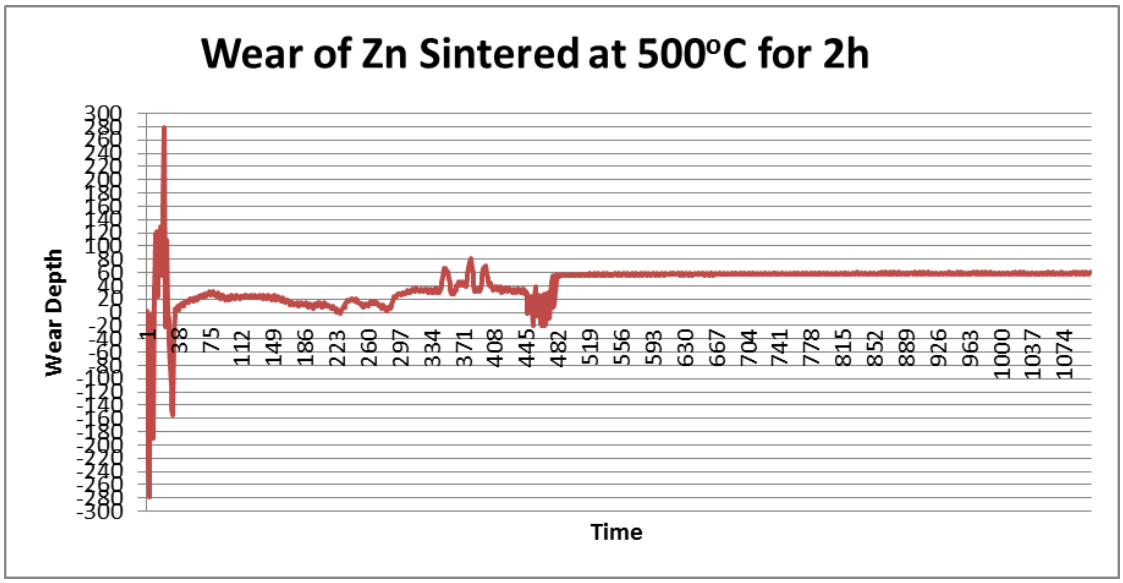


Figure 19: wear graph of Zn sinterd at 5000C

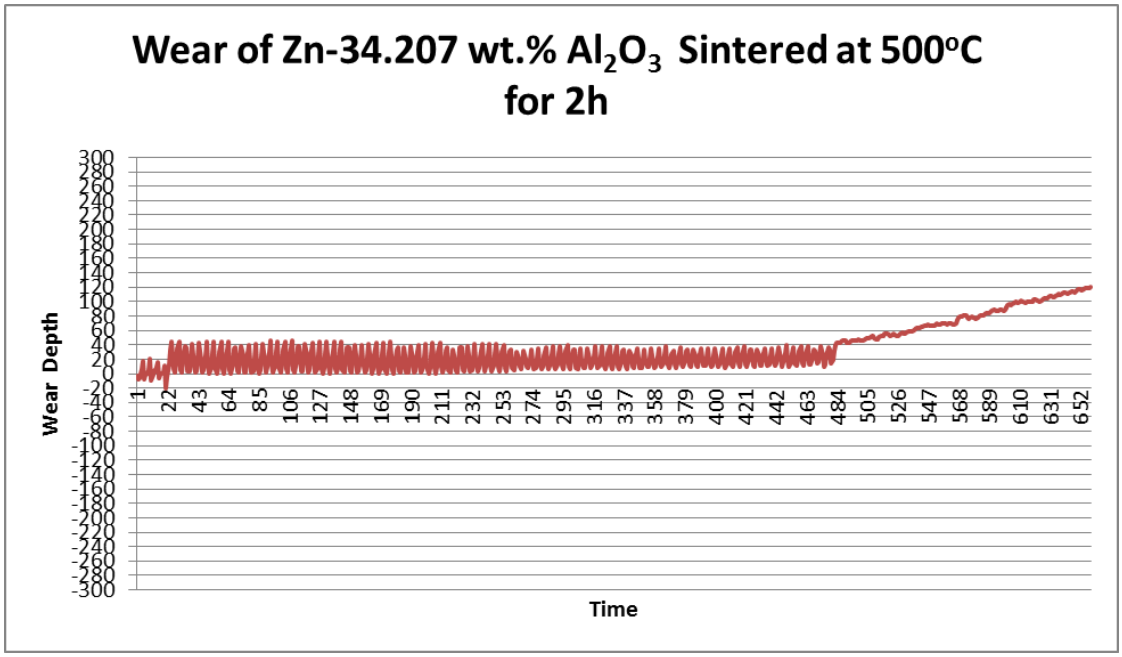


Figure 20: wear graph of Zn-48.45 vol % Al2O3 sintered at 500oC

In the case of Zn-34.207 wt.% Al_2O_3 (48.45 vol. %) composite sintered at 500°C for 2h there is a slightly lower wear as compared to Zn sintered at 500°C for 2h. The wear is almost constant throughout the period with a wear depth value of almost 40 whereas in the case of Zn sintered at 500°C for 2h sample there seem to be an increase in wear and reaches a highest value of 80 wear depth.

4.1.4.2 Wear texture: SEM images of Wear surfaces:

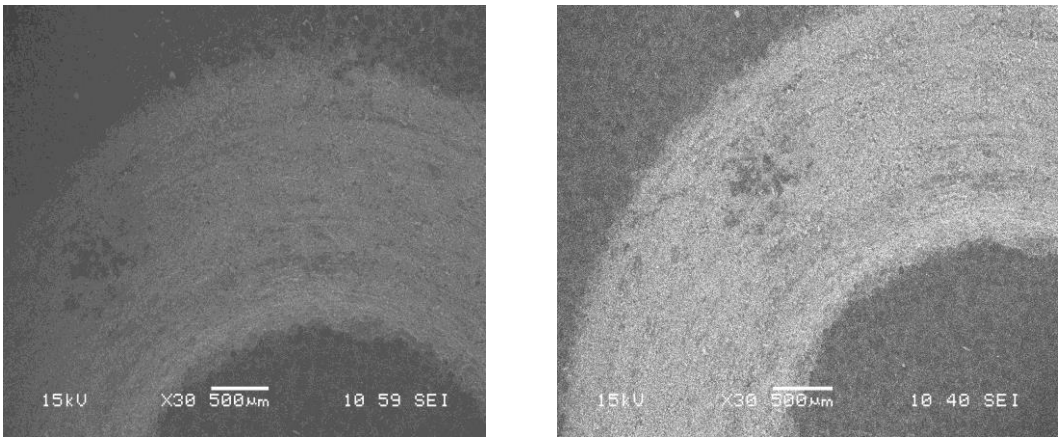


Figure 21: Wear textures (a) sintered Zn-48.45 vol. % Al_2O_3 *at 500oC for 2 h 10x (b) Zn sintered at 500oC for 2 h 10x*

As we can see that the appearance of the wear path is more profound in Zn sintered at $500^{O}C$ because of the more abrasion of the upper surface, although the appearance of the wear path of composite is dull compared to that of Zn sintered.

4.1.4.3 Study of Wear Surface:

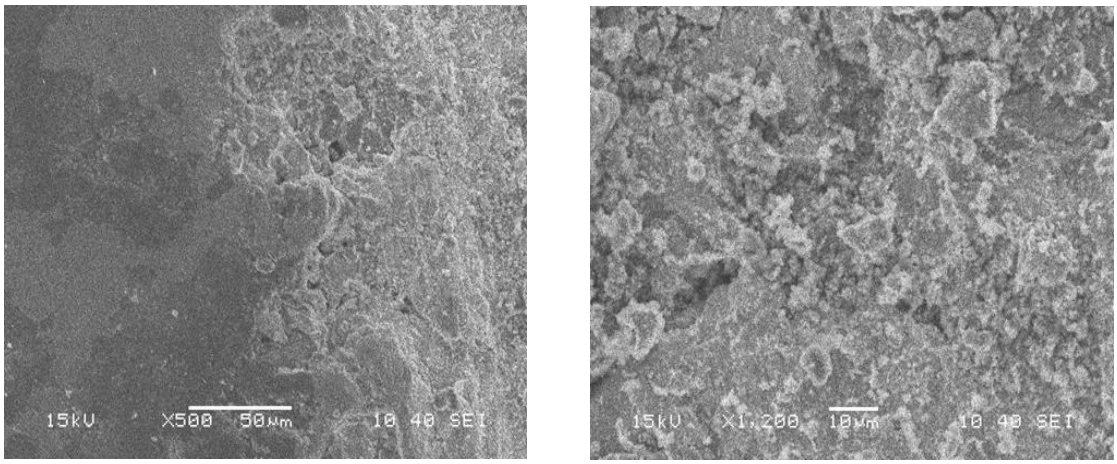


Figure 22:Wear SEM a) Zn-48.45 Al_2O_3 *sample sintered at 500 C at 500X b) at 1200X*

Here it is observed that at the wear surface, the worn out part is almost completely Al_2O_3. This may be possible because Al_2O_3 particle does not get sintered at $500^{0}C$ and these particles are loosely bonded with Zn on the upper surface and as well as with other Al_2O_3 particles. Most of the zinc particles do not get separated because they get coalesced during the sintering mechanism. Another reason of less wearing of Zn is because of its ductile property. During the wear test it gets plastically deformed but does not wear out.

4.1.5 Fracture Surface Analysis :

4.1.5.1 4.1.5.1 The fracture surface of Zn sintered at 500 C for 2 hrs:

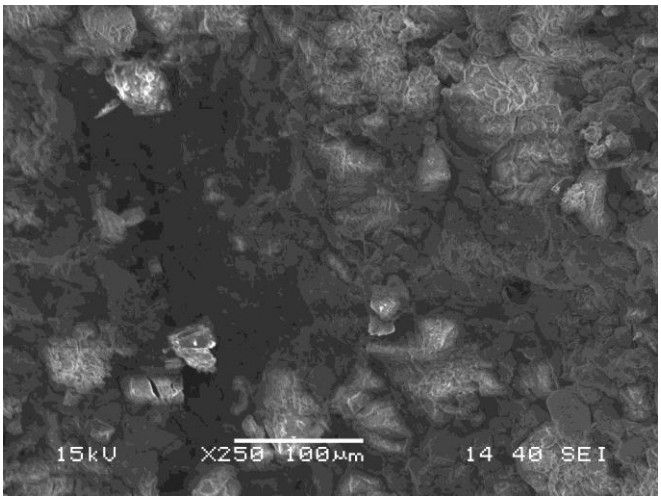
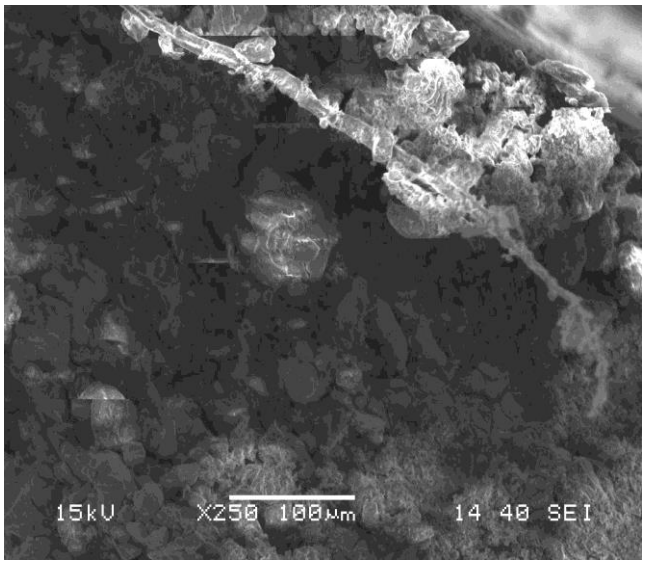


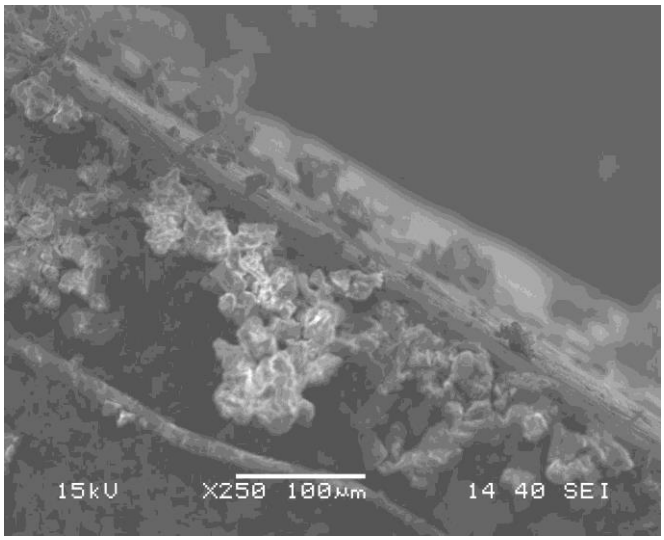
Figure 23: The fracture surface of Zn sintered at 500 C for 2 hrs

The fracture surface of Zn sintered at 500 C for 2 hrs is faceted. This shows that Zn particles looses its ductile nature during sintering. Although there is some amount of deformation on the fractured surface is observed.

4.5.1.2 Fracture surface of Zn -48.45 vol % alumina composite:



a



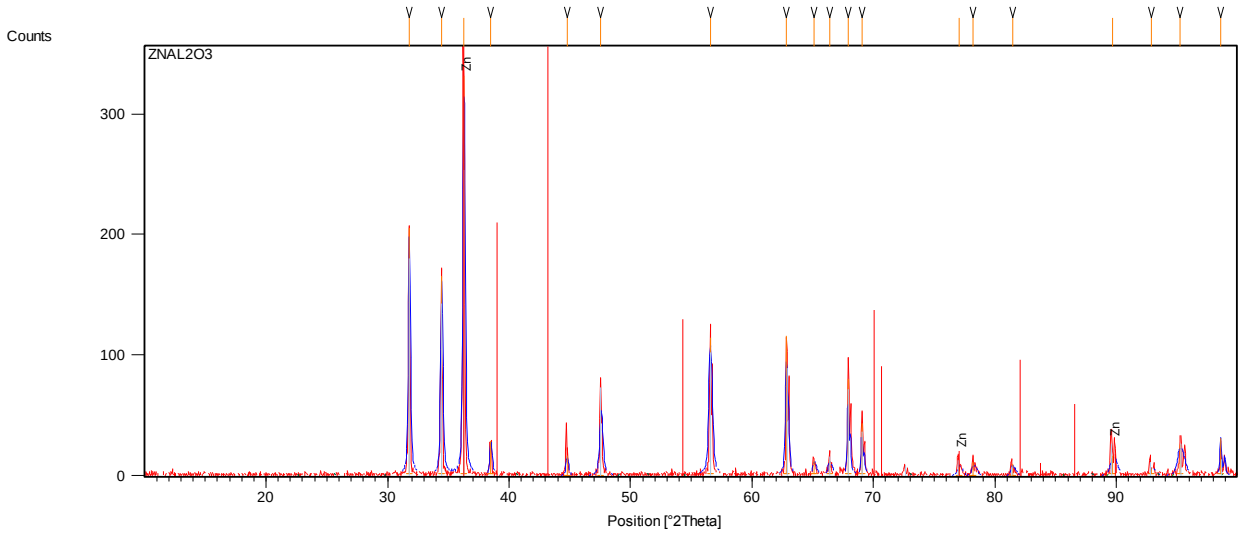
b

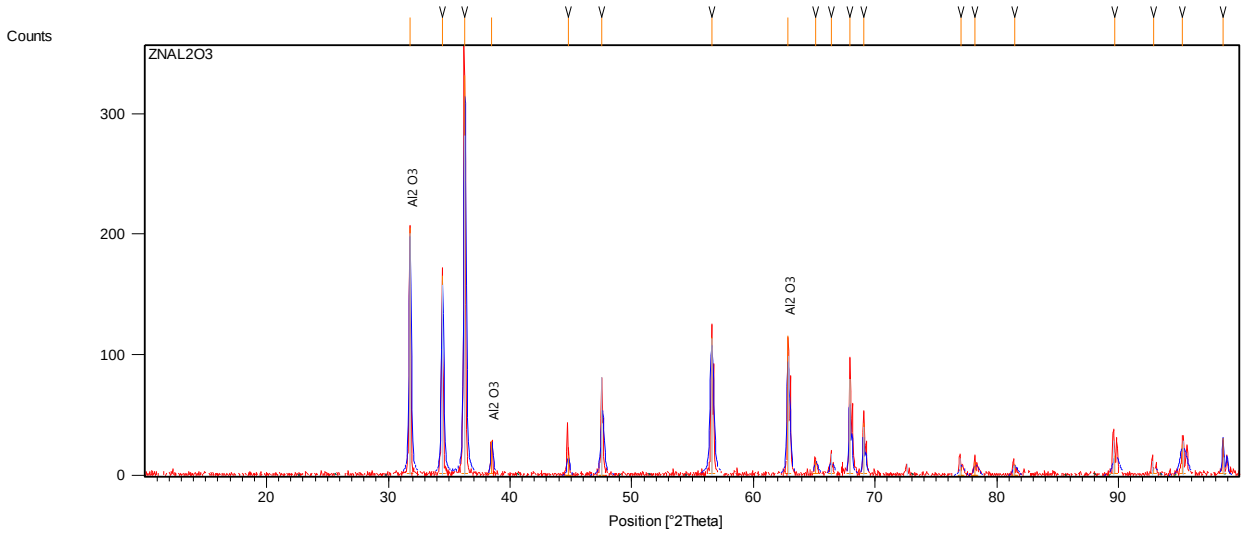
Figure 24: fracture surface of Zn -48.45 vol % alumina composite sintered at 500 C for 2 hrs a) 250X b) 250X

Though the fracture surface is not much different than that of Zn metal sinter but here alumina particles are present at the fracture surface. Due to this very reason composites imbibe greater hardness value.

4.2 Results of the sample prepared by blending of Zno and Al:

4.2.1 XRD analysis of Zn-48.45 vol % Al_2O_3:





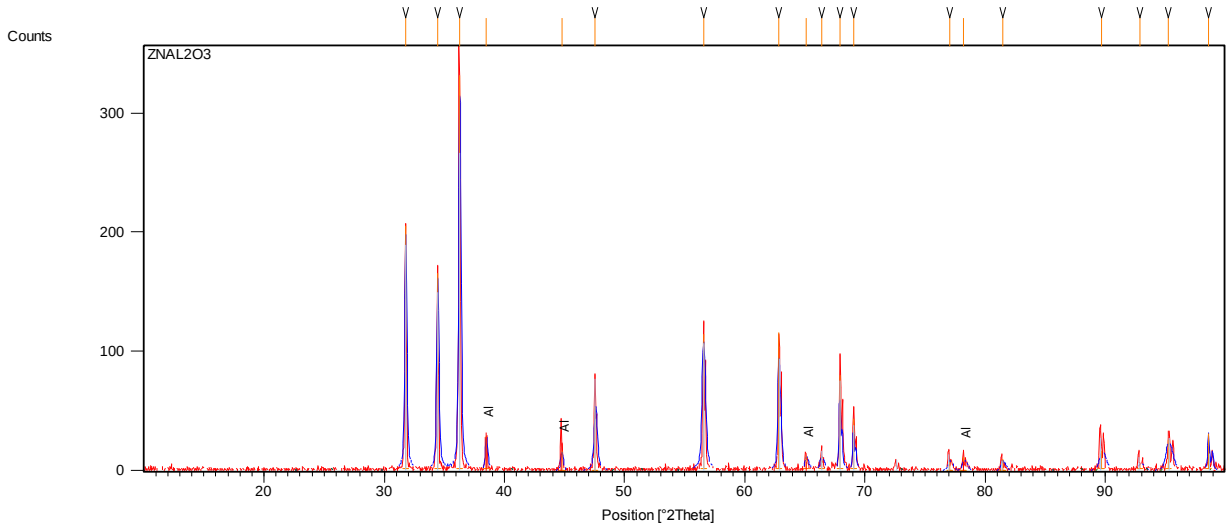
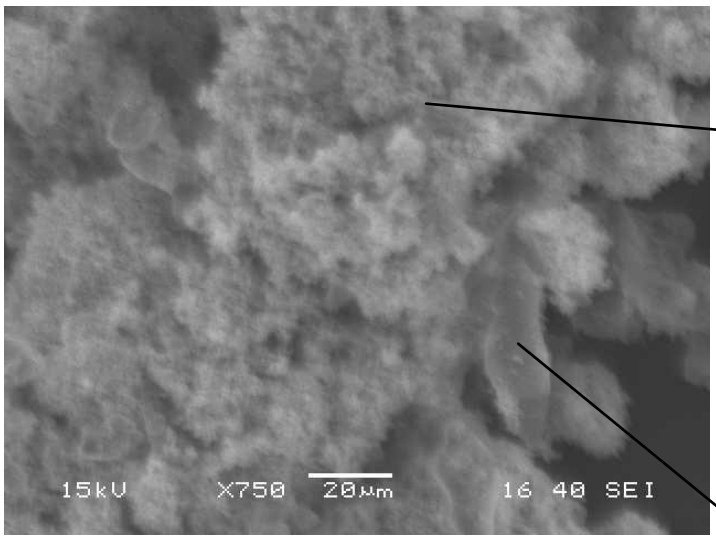


Figure 25: XRD pattern of composite

By the analysis of XRD patterns, it is found that the alumina formed is very less in percentage. There a lot of residual aluminium. The residual aluminium has not reacted with ZnO to reduce it to Zn. The conversion of Al to Al_2O_3 is not very high.

This can easily be seen in the SEM images

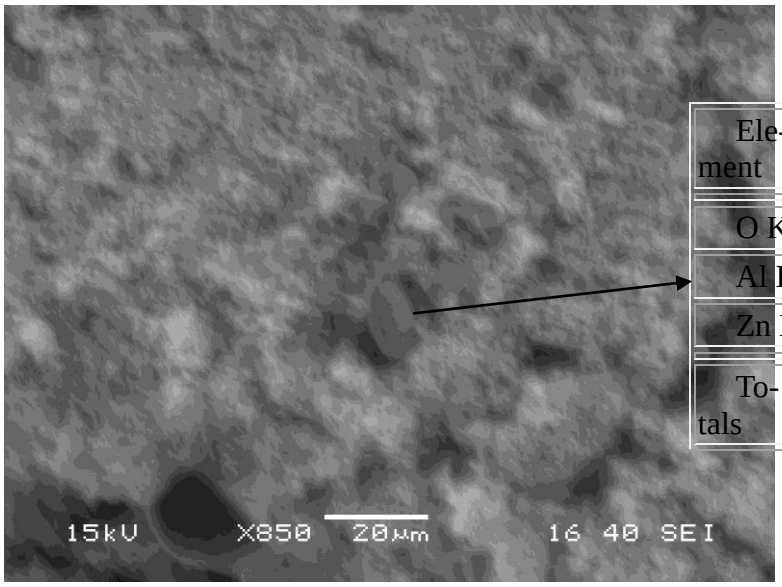
4.2.2 SEM images:



a

Element	Weight%	Atomic%
O K	52.35	72.08
Al K	24.75	20.20
Zn K	22.91	7.72
Totals	100.00	

Element	Weight%	Atomic%
O K	20.22	29.95
Al K	79.76	70.05
Zn K	0.02	0.01
Totals	100.00	



Element	Weight%	Atomic%
O K	22.78	33.34
Al K	76.52	66.41
Zn K	0.71	0.25
Totals	100.00	

b

Figure 26: SEM image of composite Zn-48.45 vol% Al_2O_3 at (a) 750X (b) 850X

4.2.3 DSC Analysis:

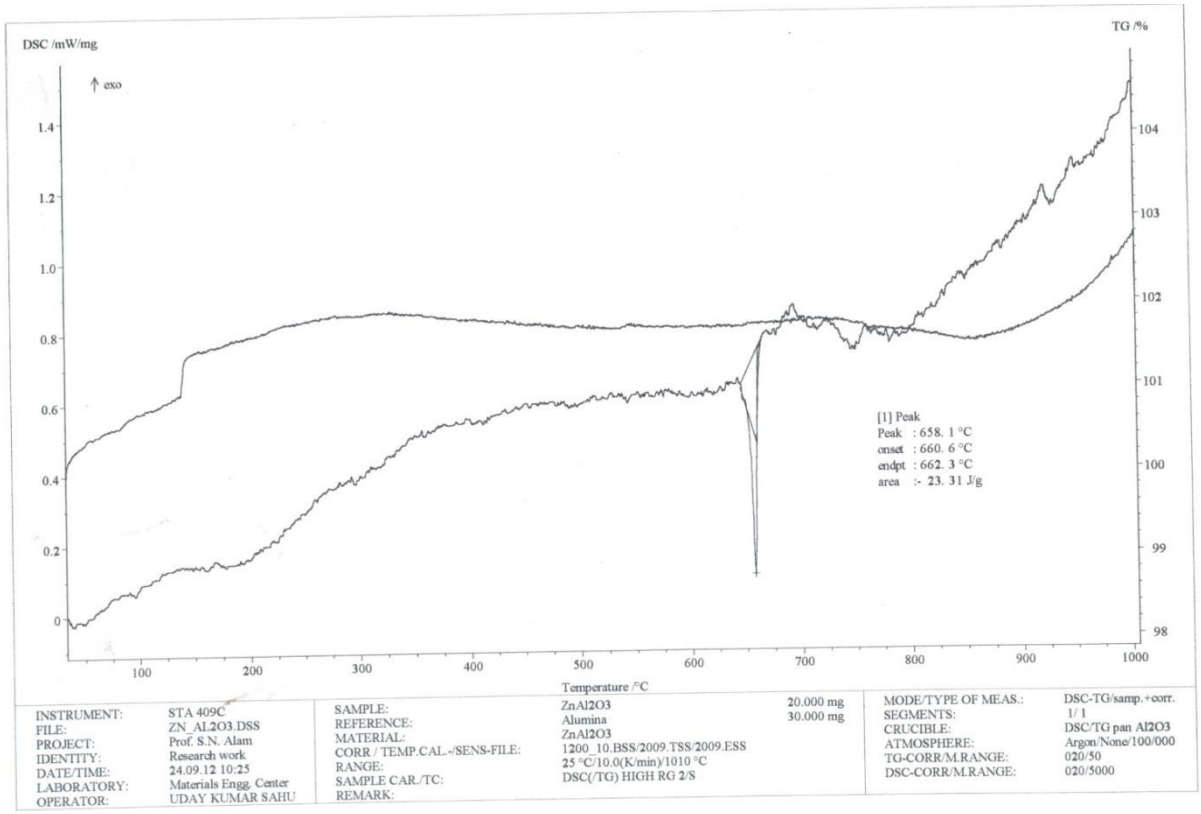


Figure 27: Thermal analysis of composite of Zn-48.45 vol. % A2O3

All the Al has not converted to Al_2O_3.Residual Al melts at 658.1^0C. The exothermic peak just after the endothermic peak is due to the crystallization of amorphous Al_2O_3. The DSC peak is due to the transformation of amorphous alumina to crystalline alumina. The metastable γ phase is converted to the more stable α phase. The crystallization of alumina gives an exothermic peak in the DSC.

Chapter 5

Conclusion

5 Conclusion:

1. By the metallothermic process a uniform distribution of Al_2O_3 can be achieved in the Zn matrix.
2. Alumina has been successfully formed by reaction of ZnO with Al leading to the formation of the Zn/ Al_2O_3 composite.
3. Improvement in hardness has been seen by adding larger volume percent of Al_2O_3 in Zn matrix.
4. Improvement in wear properties has been seen in the case of Zn/ Al_2O_3 composite.
5. Residual Al, which has not been converted to Al_2O_3, has also been found in the composite as a result it is possible to a get a Zn-Al/ Al_2O_3composite.

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